4th Quarterly Report – Public Page

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Prepared for: *PHMSA-DOT, National Biodiesel Board, Steel tank Institute, DNV Research and Innovation*

Project Title: *Corrosion and Integrity Management of Biodiesel Pipelines*

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Technical Status

Quarter Four of this research program has finally seen some tangible progress. Data has begun to be generated based on 30 day exposures of pipe steel samples in inhibited and uninhibited B100 fuel. This is the basis for the matrix of tests to be performed under the guise of Technical Task 2. Also associated with the "Inhibitors" is the investigation of the plausibility of making electrochemical measurements to monitor corrosion resistance of pipe steel samples using a micro-electrode technique. This technique allows electrochemical measurement in highly resistive media such as un-doped B100 fuel.

Task 3 involves the electrochemical monitoring of Cu-containing alloy specimens when exposed to biodiesel fuel blends. The corollary to this testing is the documentation of fuel degradation due to the incidence with the Cu-containing alloy. An array of eight test cells similar to what is being used in the inhibitors testing setup has been assembled and the delay in testing is with respect to the availability of a multiplexer for the electrochemical measurements. It is the intention of this work to begin monitoring fuel degradation in Q5 whether the electrochemical measurements can take place or not. The commencement of the study of corrosion behavior of the Cu-alloy (Alloy 360 Brass) coupons can pick up when ever equipment becomes available.

For technical tasks 2 and 3, it is still this project's intention to test three different saturation levels of biodiesel to address the differing properties of ASTM D 6751 spec fuel. Currently, however, we may be limited by the feedstock made available to us by independent suppliers, outside of the NBB, willing to donate directly to DNV to aide in the progress of our testing program. The National Biodiesel Board has still not yet been able to provide fuel samples for this testing, however Soy-based biodiesel stock has been supplied by independent sources.

Finally, for Task 4, the technical task focused on evolution of physical properties of elastomeric materials, the preliminary testing of the block oven was initiated and it was discovered that new plumbing was required to ensure that the condensation of evaporated fuel would be properly maintained. This construction effort is nearly finished and testing of elastomer samples in B100 will begin in Q5.

A summary of the three technical tasks is as follows:

Task 2 – Corrosion Inhibition Performance
Task 3 – Integrity of Non-Ferrous Metallic System Components (Cu-alloys)
Task 4 – Integrity of Non-Metallic System Components (Elastomers)

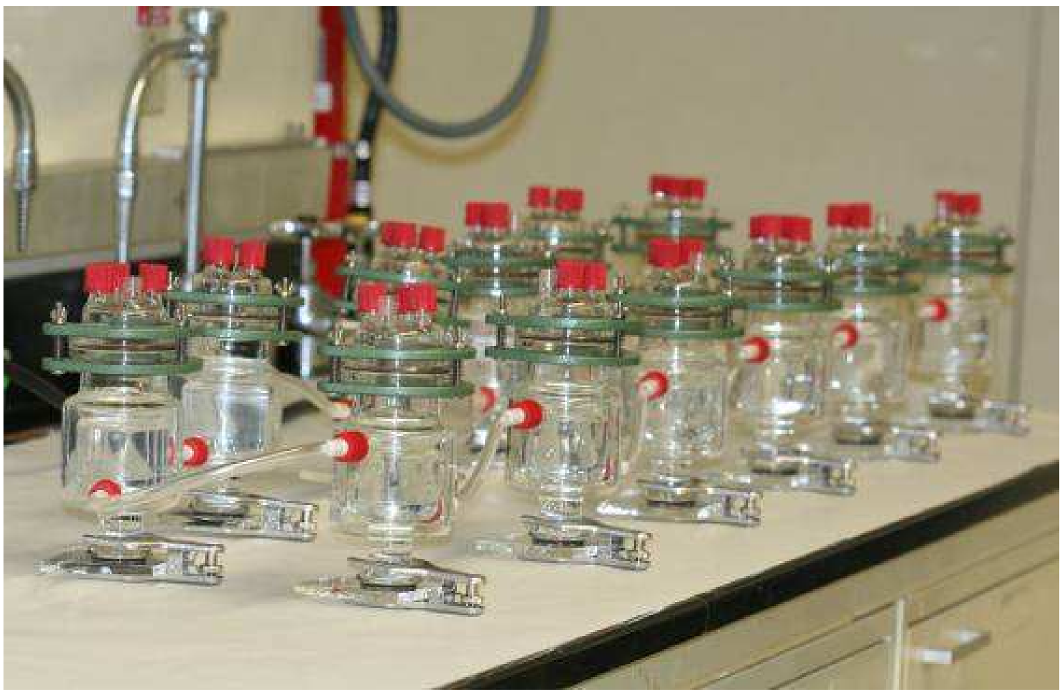

Figure 1. Array of test cells for Task 2 (inhibitors testing) in biodiesel (an array exactly similar will be used for Task 3 testing).

A close up of one of the glass cells in the area is pictured in the next figure.

Each test cell is daisy-chained to a circulating bath that keeps the immersions at 38 +/- 1ºC. 30 day immersions have been documented with neat B100 fuel as well as two dopages of Nalco EC5407A corrosion inhibitor. Samples analyzed for weight loss as well as visually inspected for pit formation on the surfaces. The results from the first sets of immersion tests are tabulated.

Inhibitor	Concentration	visual inspection (# of pits)	average weight change (g)	average corrosion rate (mpy)
N/A	0	0	11.6	1.275
	5 ppm	0	1.45	0.159
Nalco	10 ppm	0	4.85	0.533

Figure 2. typical results from inhibitors testing

The second portion of inhibitors testing has also begun in the 4th quarter. This is the exploratory electrochemical monitoring of corrosion behavior via a micro-electrode technique.

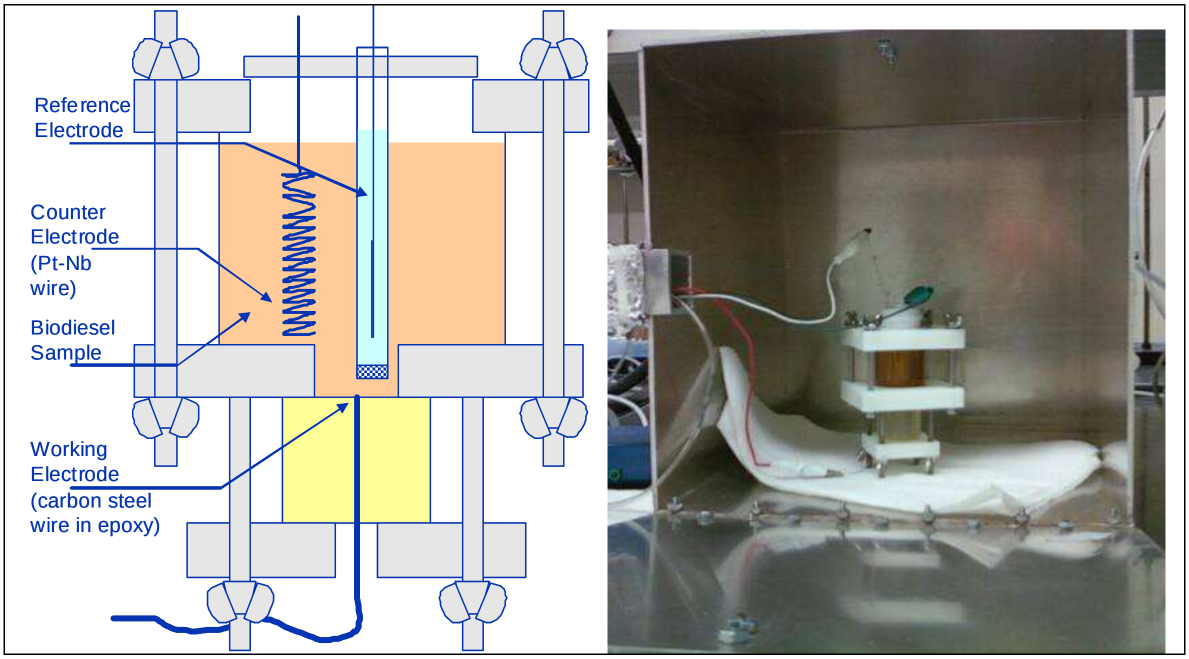

Figure 3. microelectrode test cell setup

Many preliminary tests have been conducted with constant troubleshooting to develop the best setup for utilizing this technique for corrosion behavior discernment. So far, it seems that the neat B100 is too resistive to get reproducible data. The next steps are to dope the B100 with supporting electrolytes, such as Tetrabutylammonium Tetrafluoroborate, designed to increase the conductivity of the solution but have very little effect on the corrosivity. Selected results from previous trials are presented below.

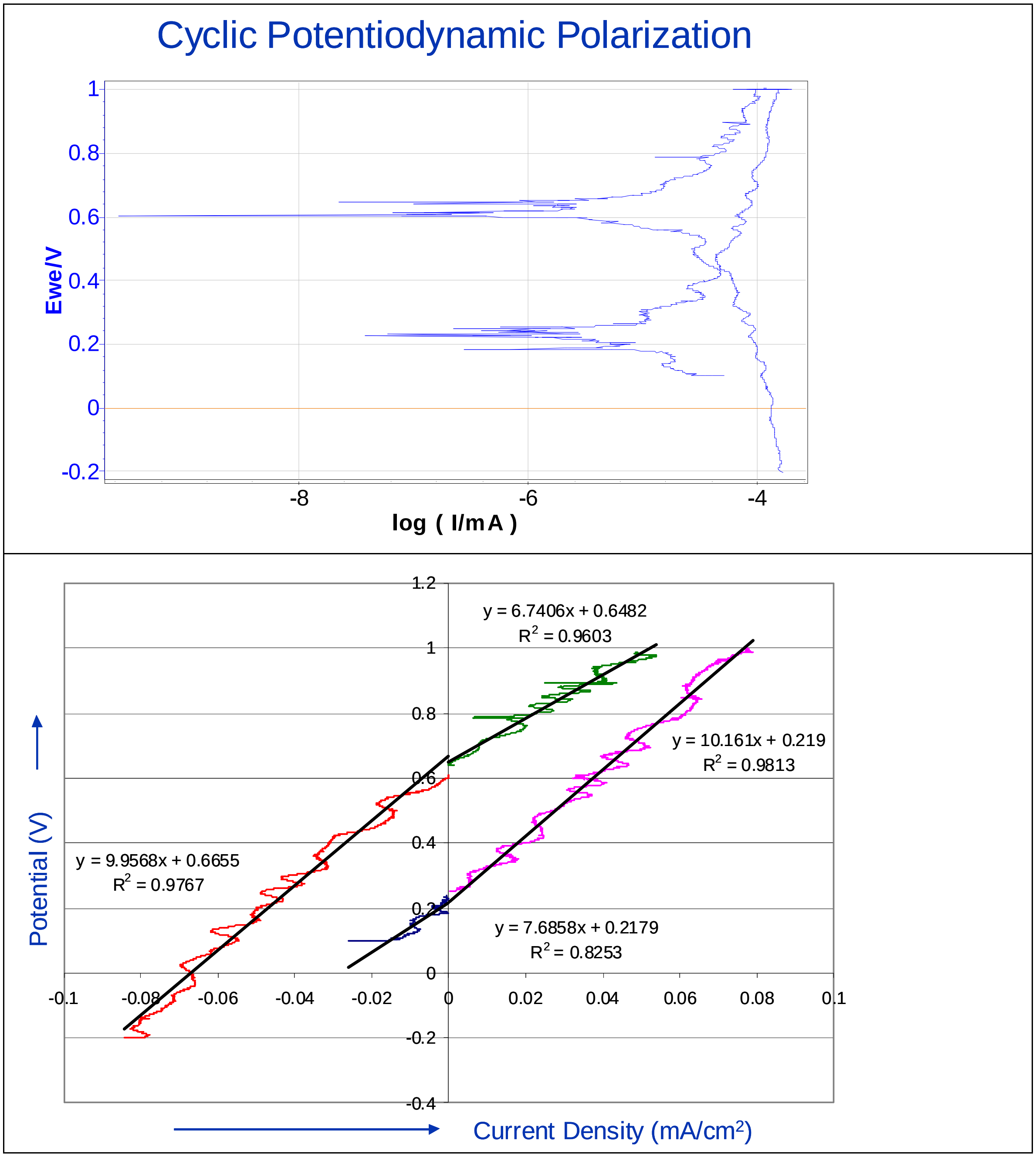

Figure 4. representative data collected for from microelectrode testing

The first plot shows a CPP curve which details corrosion behavior in a current versus potential plot. The behavior exhibited is sensible and seems to signify that not corrosion breakdown is occurring, however when the same data is plotted on a linear scale, the forward and reverse scans are depicted as straight lines which signifies that the system is acting purely as a resistor. i.e. no measurement of interaction between the sensing electrodes is being documented. This result has led us to believe that the neat B100 is not conductive enough for the sensitive electrochemical measurements to be made.

Task 2 – Integrity of Non-Ferrous Metallic System Components (Cu-alloys)

Task 2 has two main objectives which remain unchanged. The first material-related objective is to document the degradation over time of Cu-containing alloys when immersed in the various blends of biodiesel. For this, Alloy 360 will be utilized as a prudent example of common Cu-based alloys which experience regular incidence with the fuel in pump housings and seals. A series of eight additional test cells like pictured in Figure 1 will be allocated to electrochemical monitoring of the Cu-alloy corrosion behavior using a multiplexed potentiostat. The testing has not begun due to unavailability of the aforementioned multiplexer. Fuel oxidation tests will commence in Q5 with or without concurrent monitoring of Cu-alloy samples.

Task 3 – Integrity of Non-Metallic System Components (Elastomers)

For the testing of elastomer materials, a 28 test tube block furnace is used. Each test tube will hold contain one specific experiment in the matrix. Preliminary testing of the block oven pictured below signaled that the hose network needed to be re-plumbed to ensure that the composition of the test fuels will not change due to evaporation over time.

Figure 5. "Medusa", the 28 vessel block oven used for elastomeric testing in fuel.

Task 4 testing should commence in the beginning of Q5 and data will be available shortly there after.

This project unfortunately has been hindered by one setback after another, but as soon as all of the preliminary struggles are conquered, the data collection should proceed smoothly and regularly moving forward.